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Field of the invention

The invention relates to a method to the production low-viscous water-soluble polymer dispersions contained at least a polymere dispersing agent and a polymer, that by the incorporation of suitable amounts of a N-methylol-functional crosslinker predominantly only in a later processing step crosslinked. The polymer dispersions go when diluting with water into solution and become particularly as adhesives, in addition, as flocculant used.

▲ top

State of the art

Aqueous dispersions of water-soluble polymers are known. They become for example as flocculants with the waste water treatment, used as drainage means of sludge, as retention means with the papermaking, as soil improvement means or as dispersing agents.

EP 170,394 describes a liquid existing from particles, constructed from an high molecular polymer gel, which exhibits particle sizes of over 20 μm , in aqueous solution, mixable with water. The continuous phase is here an aqueous solution, a contained "equilibration means", that the water content of the gel particles with the water content in the continuous phase in the equilibrium holds and that thus an agglomeration of the gel particles prevented. As preferred equilibration means the sodium salt of the polyacrylic acid becomes and/or. Polydiallyldimethylammoniumchlorid (Poly DADMAC) used.

In EP 183,466 a method becomes the production of a water-soluble polymer dispersion described, characterized by polymerizing water-soluble monomers bottom agitations in an

aqueous solution of at least a salt in presence of a dispersing agent. Here find polyols, Polyalkylenether, alkali salts of the polyacrylic acid and alkali salts of the Poly-2-Acrylamido-2-methylpropansulfonsäure as dispersing agent use.

The DE-PS 29 24 663 covers a method to the production of an aqueous dispersion from a water-soluble polymer mass with good stability and flowability, whereby the water-soluble polymer contains at least of water-soluble, ethylenic unsaturated monomer and whereby present as dispersing agents Polyalkylenether, polyethyleneimine or other polymers can be, if necessary in presence of inorganic salts. In such a way prepared dispersion can, if necessary after diluting with water, when flocculants, thickening agent, become soil conditioning means and for other applications used.

In EP-A 262,945 an homogeneous mixture from two water-soluble polymers 1 and 2 is stressed, whereby polymer 1 becomes 2 prepared by polymerization of corresponding Monomer in the aqueous solution of polymer. As active substance concentrations, i.e. as sum contents of polymer of the 1 and 2, become related to the aqueous solution at least 10 Gew. - % mentioned. The proportions of polymer 1 to polymer 2 lie between 10: 1 and 1: 20. Field of application of such polymer solutions are the stabilization of suspended clay/tone particles against Anquellen as well as the use as flocculants and retention means with the papermaking.

EP-A 573,793 covers aqueous dispersions of water-soluble polymers formed by polymerization of a mixture existing from water-soluble, hydrophobic and if necessary amphiphilic monomers in presence of a polymere dispersing agent. The German patent applications P 43 16 200,2, P 43 35 567,6 as well as P 44 01 951,3 describe methods to the production of such dispersions of water-soluble polymers.

In the German patent application P 44 06 624,4 a method becomes the production low-viscous polymer dispersions contained at least a polymere dispersing agent, as well as a copolymer existing from at least water-soluble monomers, at least crosslinking monomers with at least two radical polymerizable groups, as well as if necessary at least hydrophobic monomers and if necessary at least amphiphilic monomers a described. The crosslinking made here exclusive over monomers with at least two radical polymerizable groups already during the polymerization.

Object and solution

With the production of the aqueous dispersions of water-soluble polymers by polymerization of hydrophilic monomers in aqueous phase, described in the state of the art, portions of cross-linking-effective substances become, z. B. crosslinking monomers avoided, since such portions lead with high polymer-held to the formation of macroscopic gels with extreme high viscosities. Such crosslinking agents are on the other hand in relative moderate portions of interesting for the generation high molecular weights with the water-soluble polymers, with which for example

high flocculation efficiency or a good effecteffect effect can become achieved.

It is an object of the invention, the crosslinking at least partially not during the polymerization to only accomplish but targeted in a later processing step. Thus the formation of macroscopic gels with extreme high viscosities in the dispersion can become avoided.

Beside the provision of aqueous dispersions of water-soluble polymers, which contain a certain proportion at crosslinking monomer units, further the object existed, high content at polymer active agent in the aqueous dispersion and simultaneous low viscosity of the aqueous dispersion to obtained. Beyond that the object covered the production of such aqueous dispersions of water-soluble polymers in absence of an oil phase and in absence of salts, like it for example from EP-A 183,466 described will, in the aqueous phase, in order to hold the portion of ecological precarious substances in the dispersions as low ones as possible.

Surprisingly that the object becomes excellent dissolved with aqueous dispersions PD) water-soluble polymers A), formed was found by the polymerization of the monomer components:

- a1) 50 to 99.99 Gew. - % at least water-soluble monomers,
- a2) 0.01-10 Gew. - %, preferably 0.01-3 Gew. - % of a N-methylol group-haltigen crosslinker, in particular N-methylol group-haltigen monomers
- a3) 0 to 1 Gew. - % preferably 0 to 0.5 Gew. - % crosslinking monomers with at least two ethylenic unsaturated, radical polymerizable groups,
- a4) 0 to 30 Gew. - %, in the particular case 1 to 20 Gew. - %, at least hydrophobic monomers, as well as if necessary
- a5) 0 to 20 Gew %, in the particular case 0.1 to 15 Gew % at least amphiphilic monomers in aqueous phase in presence of a polymere dispersing agent D).

In preferable embodiments of the invention at least one water-soluble monomers a1) at least an ionic group, is that cross-linkingable monomers a2) N-methylol acrylamide or N-methylol-methacrylamid, is that of the crosslinking monomers a3 exhibits) selected from the group that (meth) the acryl connections, trichloroethylene (meth) acryl connections, tetra (meth) acryl connections, as well as (Meth) allyl (meth) acryl connections, that is hydrophobic monomers a4) a compound of the formula I:

EMI6.1

how

G 1 for hydrogen or alkyl with 1 to 5 carbon atoms, as well as

R2 for alkyl, Cycloalkyl, aryl or Aralkyl with 1 to 20 carbon atoms or for

EMI6.2

with R3 for alkyl, Cycloalkyl, aryl, or Aralkyl with 1 to 20 carbon atoms and Z for 0, NH or NR3 to stand can,

and that is amphiphilic monomers a5) a compound of the formula II:

EMI6.3

how

A1 for O, NH, NR₄ with R₄ for alkyl with 1 to 4 carbon atoms,

R₅ for hydrogen or methyl,

R₆ for alkyls with 1 to 18 carbon atoms,

R₇ and R₈ independently for alkyl with 1 to 6 carbon atoms,

R₉ for alkyls with 1 to 6 carbon atoms,

R₁₀ für alkyl, aryl or Aralkyl with 8 to 32 carbon atoms, as well as

X for halogen, OCN, SCN, SO₄CH₃, acetate

to stand know, or a compound of the formula III:

EMI7.1

how

A₂ für O, NH or NR₁₃ with R₁₃ for alkyl with 1 to 4 carbon atoms,

R₁₁ for hydrogen or methyl,

R₁₂ for alkyl with 8 to 32 carbon atoms,

Y for alkyls with 2 to 6 carbon atoms and

n for a whole number between 1 and 50

to stand can.

In an other preferable embodiment of the invention the polymer A becomes) in presence at least a polymere dispersing agent D), as described, prepared and the resultant polymer dispersion PD) thereafter in a second stage other dispersing agent D) in aqueous solution added in a first stage (see. Also DE-patent application P 43 16 200,2). It results the polymer dispersion PD min).

Further 43 35 567,6 of the production of the polymer dispersion PD can follow) or the polymer dispersion PD min) in a first stage the reduction of the water content to a concentrated polymer dispersion as well as in a second stage the addition of other dispersing agent D) in aqueous solution in accordance with the German patent application P. It results the polymer dispersion PD min min).

Carrying out the invention

Monomers the a1)

As monomers a1) for example salts can become from the acryl and/or the methacrylic acid of the general formula IV used:

EMI8.1

how

R min for hydrogen or methyl and

Q (+) for alkali metal ions, as for example well (+) or K (+),

Ammonium ions as for example NH₄ (+), (+) NR min min H₃, (+) NR min min 2H₂, (+) NR min min 3H or (+) NR min min 4 with R min min = alkyl with 1 to 6 carbon atoms or other univalent, positive loaded ions,

to stand can.

To monomers a1) to the formula IV for example sodium (meth) belongs to acrylate, potassium (meth) acrylate or ammonium (meth) acrylate.

Furthermore for example the acrylic acid and/or the methacrylic acid as well as (Meth) acrylamides of the formula V used can become as monomer component a1):

EMI9.1

how

R< III> for hydrogen or methyl, as well as

R< IV> and R< V> independently for hydrogen and/or for if necessary functionalised alkyl radicals with 1 to 5 carbon atoms to stand can.

As monomers a1) of the formula V are exemplarily mentioned:

(Meth) acrylamide, N-methyl (meth) acrylamide, N, N-Dimethyl (meth) acrylamide, N-methyl-N-ethyl (meth) acrylamide as well as n (2-Hydroxy) ethyl (meth) acrylamide.

To the production (Meth) of the acrylamides see. for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd. OD., volume. 15, sides 346 to 376, Wiley Interscience, New York, 1981.

Further monomers of the formula VI used can become as monomer component a1):

EMI10.1

how

R< IV> for hydrogen or methyl,

Z1 for 0, NH or NR4, as well as

L for the groups:

EMI10.2

whereby L1 and L4 for an alkylene radical or hydroxyalkyl remainder with 2 to 6 carbon atoms, L2, L3, L5, L6 and L7 independently for hydrogen or an alkyl radical with 1 to 6 carbon atoms, as well as Z for halogen, acetate or SO₄CH₃ to stand can.

For monomers a1) of the formula VI mentioned are exemplary:

2 (N, N-Dimethylamino) ethyl (meth) acrylate, 3 (N, N-Dimethylamino) propyl (meth) acrylate, 4 (N, N-Dimethylamino) butyl (meth) acrylate, 2 (N, N-Diethylamino) ethyl (meth) acrylate, 2-Hydroxy-3 (N, N-dimethylamino) propyl (meth) acrylate, 2 (N, N, N-trimethylammonium) ethyl (meth) acrylate chloride, (3 (N, N, N-trimethylammonium) propyl (meth) acrylate chloride or 2-Hydroxy-3 (N, N, N-trimethylammonium) propyl (meth) acrylate chloride and/or. (Meth) the acrylamides of the above mentioned. Compounds, as for example 2-Dimethylaminoethyl (meth) acrylamide, 3-Dimethylaminopropyl (meth) acrylamide or 3-Trimethylammoniumpropyl (meth) acrylamide chloride. To the representation (Meth) of the acryl ammonium salts see. for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd. OD., volume. 15, sides 346 to 376,

Wiley Interscience, New York, 1981. Als monomer component a1) can become further ethylenic unsaturated monomers, which are capable to the formation of water-soluble polymers, used, as for example vinyl pyridine, N-vinylpyrrolidone, styrene-sulfone-acidic or N-vinylimidazole.

Beyond that also combinations are various, bottom a1) of aforementioned water-soluble monomers possible.

The N-methylol group-haltigen crosslinking agents a2)

The incorporation of N-methylol group-haltigen crosslinking agents into the polymer dispersions according to invention is an essential invention characteristic. Crosslinkers on N-Methylolbasis are known from the lacquer production. The crosslinking taking place here has the advantage that it targeted by outside action, z. B. by acidic additive and/or by temperature influence effected will can. Usually made it in drafts of the film formation. In the polymer dispersions according to invention it can be made partially via a "Nachpolymerisation" with a temperature higher to 40 degrees C by 0,1-10 hours. Conventional ones are 5-15 degrees C temperature rise and 0.5-3 hours. The "Nachpolymerisation" serves however mainly the reduction residual monomers.

An application is provided, where a film formation takes place, z with the polymer dispersion according to invention. B. with use as wallpaper coating, a so made to a large extent complete crosslinking with jobs on the underlay by drying bottom if necessary brief application of an high temperature. By the low product viscosity it is possible to lay the polymer dispersion on also in thin layer z. B. < 10 g dry weight of the Films/m< 2>. The high molecular weights an involve high cohesion of the adhesive film after made application, generated by the crosslinking.

N-methylol groups possible with itself to react (own cross-linking) or also with other functional groups as amide groups or hydroxyl groups (foreign cross-linking). Both possibilities are in the instant invention given. For foreign cross-linking are suitable functional groups the bottom monomers a1, z. B. Acrylamide.

The crosslinking agents according to invention are condensation products of formaldehyde with amides or amines. They contain or several N-methylol groups, also as N-hydroxymethyl groups referred. Preferred ones are monomers with N-methylol groups, like N-Methylolmethacrylamid or N-methylol acrylamide. With their incorporation develop polymers, which contain cross-linkingable N-methylol groups in the polymer chain. Suitable ones are all monomers, which contain both an ethylenic unsaturated, radical polymerizable group, and or of several N-methylol-functional groups.

Added can work become instead of its in addition, not polymerizable compounds, which contain 2 or several methyl oil groups and thus as crosslinkers for the hydrophilic polymer chains a1)

can. Suitable ones are here z. B. Dimethylolharnstoff, Trimethylolmelamin, Hexamethylolmelamin, or low molecular, water-soluble melamine resins. These cross-linking compounds do not become incorporated during the polymerization or only in subordinate mass into the polymer chain. They become partially with the Nachpolymerisation cross-linking-effective, react however to a large extent complete as crosslinkers with a later processing step such as z. B. drying Films. Die N-methylol group-haltigen crosslinking agents a2) become forwards or during the polymerization in amounts of 0,01-10 Gew. - %, preferred 0.01-3 Gew. - %, particularly preferred 0.02-2 Gew. - % added.

Crosslinking monomers the a3)

As other crosslinking agents in the form of crosslinking monomers compounds with at least two polymerizable groups use find. For compounds with two radical polymerizable, ethylenic unsaturated groups can stand:

1) Alkenyldi (meth) of acrylates as for example 1,6-Hexandioldi (meth) acrylate, 1,10-Decandioldi (meth) acrylate, 1,12-Dodecandioldi (meth) acrylate, 1,18-Octadecandioldi (meth) acrylate, Neopentylglycoldi (meth) acrylate, Methylendi (meth) acrylate, 2,2 min - to (hydroxymethyl) - 1,3-propandioldi (meth) acrylate, as well as preferably Glycoldi (meth) acrylate, 1,3-Propandioldi (meth) acrylate, 1,3-Butandioldi (meth) acrylate and 1,4-Butandioldi (meth) acrylate.

2) Alkenyldi (meth) of acrylamides as for example N-Methyldi (meth) acrylamide, N, N min -3-Methylbutylidenbis (meth) acrylamide, N, N min - (1,2-Dihydroxyethylen) to (meth) acrylamide, as well as preferably N, N min - Hexamethylenbis (meth) acrylamide and particularly preferred N, N min - untiluntil until (meth) acrylamide.

3) Polyalkoxydi (meth) of acrylates of the formula VII:

EMI14.1

how

R13 for hydrogen or methyl,

m for a whole number between 2 and 6, as well as

p for a whole number between 2 and 50

to stand can.

Mentioned are exemplary:

Polypropylenglycoldi (meth) of acrylates ($m = 3$) with p between 4 and 25, Polybutylenglycoldi (meth) acrylate ($m = 4$) with p between 5 and 40, as well as those preferred used

Polyethylenglycoldi (meth) of acrylates ($m = 2$) with p between 2 and 45, as for example Diethylenglycoldi (meth) acrylate, triethylene glycol (meth) acrylate, Tetraethylenglycoldi (meth) acrylate or preferably Polyethylenglycoldi (meth) of acrylates with p between 5 and 20.

4. Als (meth) the acrylates can become for example further used:

Benzylidendi (meth) acrylate, bisphenol A those (meth) acrylate, 1,3-Di (meth) acryloyloxy-propanol-2, Hydrochinondi (meth) acrylate, Thioethylenglycoldi (meth) acrylate,

Thiopropylenglycoldi (meth) acrylate, Thiopolyethylenglycoldi (meth) of acrylates as well as

Thiopropylenglycoldi (meth) of acrylates.

5. Divinylverbindungen as for example 1,4-Butandioldivinylether or divinylbenzene, butadiene or 1,6-Hexadien, which (meth) allyl connections as for example (meth) allylphthalat or which (meth) allylsuccinat, vinyl (meth) acryl compounds as for example vinyl (meth) acrylate or preferred (Meth) allyl (meth) acryl compounds as for example allyl (meth) acrylate.

For compounds with 3 or more ethylenic unsaturated, radical polymerizable groups are exemplarily mentioned: Glycerintri (meth) acrylate, Trimethylolpropantri (meth) acrylate, Trimethylolpropantriethoxytri (meth) acrylate, Trimethacrylamid, (Meth) allylidendi (meth) acrylate, 3-Allyloxy-1,2-propandioldi (meth) acrylate, tri allyl cyanogen urate or tri allyl ISO cyanogen urate, as members for compounds with more than 3 ethylenic unsaturated, radical polymerizable groups: Pentaerythrittetra (meth) acrylate or N, N, N min, N min - tetra (meth) acryloyl-1,5-pentandiamin.

Crosslinking monomers the a3) become in combination with a2) in minor amounts of 0-1 Gew. - % related to the Gesamtkomonomeren used.

Preferred ones are 0-0.5 Gew. - %. Their addition is not compelling. If one omits it, a2 take over) the exclusive cross-linking function.

Hydrophobic monomers the a4)

Hydrophobic monomers are preferred monomers of the formula I:

EMI16.1

how

R< 1> for hydrogen or alkyl with 1 to 5 carbon atoms and

R2 for alkyl, Cycloalkyl, aryl or Aralkyl with 1 to 20 carbon atoms or for

EMI16.2

with Z for O, NH or NR3 as well as R3 for alkyl, Cycloalkyl, aryl or Aralkyl with 1 to 20 carbon atoms or for aryl with 6 to 12 carbon atoms

to stand can.

For monomers of the formula I mentioned are exemplary:

Styrene, - methyl styrene, p-methyl styrene, p-vinyltoluene, Vinylcyclopentan,

Vinylcyclohexan, Vinylcyclooctan, propylene, Buten-1, isobutene, 2-Methylbuten-1,2

Methylhexen-1,2-Propylhexen-1, methyl (meth) acrylate, ethyl (meth) acrylate, Propyl (meth) acrylate, Isopropyl (meth) acrylate, Butyl (meth) acrylate, Pentyl (meth) acrylate, Hexyl (meth) acrylate, Octyl (meth) acrylate, Decyl (meth) acrylate, Dodecyl (meth) acrylate, Tetradecyl (meth) acrylate, hexadecyl (meth) acrylate, Octadecyl (meth) acrylate, Eicosyl (meth) acrylate, Cyclopentyl (meth) acrylate, cyclohexyl (meth) acrylate, Cyclooctyl (meth) acrylate, Phenyl (meth) acrylate, 4-Methylphenyl (meth) acrylate or 4-Methoxyphenyl (meth) acrylate.

Furthermore used can become as hydrophobic monomers a4): Ethylene, vinylidene chloride, vinylidene fluoride, vinyl chloride or other, predominant araliphatic compounds with polymerizable double bonds. Also combinations are various hydrophobic Monomerer a4) possible.

Amphiphilic monomers the a5)

Amphiphilic monomers a5) can be for example monomers compounds of the formula II:
EMI17.1

how

A1 for 0, NH, NR₄ with R₄ for alkyl with 1 to 4 carbon atoms,
R₅ for hydrogen or methyl,
R₆ for alkyls with 1 to 18 carbon atoms,
R₇ and R₈ independently for alkyl with 1 to 6 carbon atoms,
R₉ for alkyls with 1 to 6 carbon atoms,
R₁₀ for alkyl, aryl or Aralkyl with 8 to 32 carbon atoms and
X for halogen, OCN, SCN, SO₄CH₃ or acetate
to stand can.

Further amphiphilic monomers the a5 covers) compounds of the formula IIa.

EMI18.1

how

A₁, R₅, R₆, R₇, R₈ and X the same importance as in formula II possess, as well as for R min 9 for alkyl with 1 to 18 carbon atoms to stand can.

For monomers of the formula II and IIa mentioned are exemplary:

EMI18.2

EMI19.1

To the production amphiphilic monomers of the formulas see II and IIa. for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd. OD., volume. 1, sides 330 to 354, volume. 15, sides 346 to 376, 1978 and 1981, Wiley Interscience, New York.

Preferably amphiphilic monomers become a5) of the formulas III and IIIa used:

EMI19.2

how

A₂ for 0, NH or NR₁₃ with R₁₃ for alkyl with 1 to 4 carbon atoms,
R₁₁ for hydrogen or methyl,
R₁₂ for alkyl, aryl or Aralkyl with 8 to 32 carbon atoms,
R min 12 for hydrogen or alkyl with 1 to 4 carbon atoms,

R14 for alkyls with 1 to 18 carbon atoms,
Y for alkyls with 2 to 6 carbon atoms, as well as
n for a whole number between 1 and 50 to stand can.

For monomers of the formulas III and IIIa mentioned are exemplary:
EMI20.1

Also combinations are various amphiphilic Monomerer a5) possible.

The polymere dispersing agent D)

The polymere dispersing agent D) differs significant in the chemical composition and in the average molecular weight M_w (weight average) of the water-soluble polymer A) and is with this incompatible. The average molecular weights M_w of the polymere dispersing agents D) lie in the range between 10^3 to 5×10^5 Dalton, preferably between 10^4 to 4×10^5 Dalton (to the determination of M_w see. H.F. Mark of et al. Encyclopedia OF polymer Science and Technology, volume. 10, sides 1 to 19, J. Wiley, New York, 1987).

The polymere dispersing agents D) contain functional groups selected from ether, hydroxyl, carboxyl, sulfone, sulfate ester, revision modification NO, revision modification DO, Imino, third. - Revision modification-NO and/or quaternary ammonium groups.

For polymere dispersing agent D) mentioned are exemplary: Cellulose derivatives, polyethylene glycols, polypropylene glycols, copolymers from ethylene glycols and propylene glycols, polyvinyl acetate, polyvinyl alcohol, starch and starch derivatives, dextran, polyvinyl pyrrolidone, Polyvinylpyridin, polyethyleneimine, Polyvinylimidazol, Polyvinylsuccinimid, Polyvinyl-2-methylsuccinimid, Polyvinyl-1,3-oxazolidon-2, Polyvinyl-2-methylimidazolin, as well as copolymers, which can contain the for example subsequent monomer units of above-mentioned polymers beside combinations from monomers devices: Maleic acid, maleic anhydride, fumaric acid, itaconic acid, Itakonsäureanhydrid, (Meth) acrylic acid, salts (Meth) of the acrylic acid or (Meth) acrylamides.

Prefered ones become as polymere dispersing agent D) Polyalkylenether as for example polyethylene glycols, polypropylene glycols or Polybutylen-1,4-ether used (to the production of Polyalkylenethern for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd compares. OD. Volume. 18, sides 616 to 670, Wiley Interscience, New York, 1982).

Particularly prefered becomes as polymere dispersing agent D) polyelectrolytes used, as for example polymers, contained salts (Meth) of the acrylic acid as anionic monomer components or as cationic devices with methyl chloride of quaternierte derivatives of N, N-Dimethylaminoethyl (meth) acrylate, N, N-Dimethylaminopropyl (meth) acrylate, N, N-Dimethylaminopropyl (meth)

acrylamide or N, N-Dimethylaminohydroxypropyl (meth) acrylate. To the production of polyelectrolytes see, for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd. OD., volume. 18, sides 495 to 530, Wiley Interscience, New York 1982.

Whole particularly preferred used becomes Polydiallyldimethylammonium chloride (Poly DADMAC) with an average molecular weight M_w between 5×10^4 and 4×10^5 Dalton as polymere dispersing agent D) used.

Furthermore low molecular emulsifiers with a molecular weight M_w can $< 10^3$ Dalton in amounts from 0 to 5 Gew. - %, in the particular case in amounts from 0,1 to 5 Gew. - % related to the entire polymer dispersion used become.

The production of the aqueous polymer dispersions contained polymer A) and polymere dispersing agent D).

The single-step production process (polymer dispersions PD))

The amount of the used monomer mixture a1), a2) as well as if necessary a3), a4) and a5) related to 100 Gew. - Lies parts waters as reaction medium between 5 and 80 Gew. - Parts, preferably between 10 and 50 Gew. - Parts. Monomers if the a1 becomes) and if necessary a5) as aqueous solution used, then the water content the reaction medium is slammed shut. The amount of the polymere dispersing agent D) related to 100 Gew. - Is appropriate parts waters as reaction medium between 1 and 50 Gew. - Parts, preferred between 2 and 40 Gew. - Parts, particularly preferred between 5 and 30 Gew. - Parts.

Generally the polymere dispersing agent in the aqueous reaction medium presented becomes before monomers the a1), a2) as well as if necessary. a3), a4) and a5) added become.

The targeted adjustment of the molecular weight the monomer mixture also a regulator can become added. This lowered the molecular weight of the polymer A), possible however the provision of particularly low-viscous polymer dispersions. Conventional water-soluble polymerization automatic controllers are Thioglykolsäure, 2-Mercaptoäthanol, Pentaerythrit tetra thioglycolat in amounts of 0.01-2%. They can be metered to beginning of polymerization at one time or successive during the polymerization.

Starting the polymerization for example radical initiators (= polymerization initiator) or high-energy radiation, as for example UV light, become used. As radical initiators preferred become for example 2,2 min - azobisisobutyronitrile, 2,2 min - Azobis (2 - amidopropan) dihydrochlorid dissolved in dimethylformamide, potassium persulfate, ammonium persulfate, hydrogen peroxide, if necessary in combination with a reducing agent, as for example an amine or a sodium sulphite, used. The portion of initiator, related to the monomer mixture a1), a2) as well

as if necessary a3), a4) and a5), is appropriate for ordinary between 10< -5> and 5 Gew. - %, preferably between 10< -4> and 1 Gew. - %, whereby the initiator can become partial beginnings of the reaction complete or added with subsequent dosage about the entire polymerization process. Just as the monomer mixture a1 can do), a2) as well as if necessary a3), a4) and a5) complete at the beginning of the polymerization or than inlet about the entire polymerization process added become partial. It has itself preserved to only add with adiabatic polymerization process a part monomers of the a2) at the temperature maximum. Thereby the viscosity of the polymer dispersion remains particularly low, the cross-linking possibility in one of the subsequent method steps is however full given. The polymerization temperature amounts to preferred between 30 and 70 degrees C, between 0 and 100 degrees C. Preferably bottom inert gas atmosphere, for example bottom nitrogen atmosphere polymerized becomes. The final conversion of the polymerization lies over 97 Gew. - % of the monomer mixture a1), a2) as well as if necessary a3), a4) and a5), for which generally 1 to 10 hours polymerization duration required are. Preferred elevated one against end of the polymerization again the temperature around 5-15 degrees C and adds other initiator. Thereby the remainder monomer content of bottom 0.1% sinks. This other initiator addition can amount to quantitatively the 2-20fache in relation to the polymerization start. Polymer dispersions PD result).

The two-stage production process (polymer dispersions PD min))

The two-stage production process covers the addition of other polymere dispersing agents D) in aqueous solution to the dispersion PD) of the polymer A prepared after the single-step method). Polymer dispersions PD min result).

For the mixing operation both static and dynamic mixers can become used. While first work by generation of turbulence, which develops in the liquid mixtures when flowing through the mixers, the turbulence becomes active generated with dynamic mixers (see. for example for this Römpps chemistry encyclopedia, 9. Aufl., side 2805, George Thieme, Stuttgart, New York, 1992).

Preferred ones become as mixer agitator used, which during the agitating procedure a small Schergefälle produces, as for example propeller, slanting sheet, disk, Impeller, kreuzbalken, lattice, anchor, schraubenspindel or spiral agitators (see. for example for this Römpps chemistry encyclopedia, 9. Aufl., sides 3939 to 3940, George Thieme, Stuttgart, New York, 1993). During the mixing operation the polymer dispersion PD prepared after the single-step method becomes preferably) presented and afterwards the aqueous solution of the polymere dispersing agent D) bottom agitations gradual added. The viscosity of the mixture becomes continuous controlled.

In one particularly preferable embodiment of the invention will the polymer dispersion PD) on 30 to 90 degrees C, preferably on 40 to 70 degrees C heated, in order to hold the viscosity during the mixing operation if possible low. A preferred Nachpolymerisationsphase becomes connected also with elevated temperature bottom addition of other initiator here.

The modified two-stage production process (polymer dispersions PD min min))

The modified two-stage production process covers the reduction of the water content of the polymer dispersions PD prepared after the single-step method in the first stage) and/or. the water content after the before described two-stage method prepared of the polymer dispersions PD min) and afterwards in the second stage the addition of polymere dispersing agent D) in aqueous solution, like with the two-stage method described. Polymer dispersions PD min min result).

The water content of the polymer dispersions PD) and/or. PD min) becomes preferably reduced by evaporation of the water. This can take place for example via Abdestillieren of the water, preferably with negative pressure or in the vacuum. The here used distillation apparatuses are known, as for example distillation columns (see. for example Kirk Othmer, Encyclopedia OF Chemical Technology, 3rd. OD., volume. 7, sides 849 to 891, Wiley Interscience, New York, 1979). Other evaporation aggregates are for example convection evaporators or thin section evaporators (see. for example for this Kirk Othmer, locomotive CIT., volume. 9, sides 472 to 493, Wiley Interscience, New York, 1980).

Also methods such as diaphragm diffusion or napkins of water with inorganic or organic reagents are possible.

The water content of the polymer dispersions PD) and/or. PD min) can become in that mass reduced, how the viscosity of the resultant polymer dispersions permits a technologically reasonable handling and as long as the dispersion stability permits it. Generally polymer dispersions with an active substance content of up to 50 Gew are in the first stage. - % related to the dispersion or light over it possible.

The addition of the polymere dispersing agent D) in the second stage of the modified two-stage production process made as with the two-stage production process described. Again will in stage 1 prepared the polymer dispersion with reduced water content preferably on 30 to 90 degrees C, particularly preferred on 40 to 70 degrees C heated, in order to hold the viscosity with adding the polymere dispersing agent if possible low. The polymer dispersions PD min min result) with very high active substance-held with comparatively very low viscosities.

Advantageous effects of the invention

Those the crosslinking monomer units a2) and if necessary a3) contained polymer dispersions PD), PD min) as well as PD min min) are characterised, measured at the active substance concentration, by a surprising low viscosity, whereby the active agent from the combination of polymer A) and polymere dispersing agents D) exists. This is the surprising, since the molecular

weights of the water-soluble polymer A) by the crosslinking monomer components a2) and if necessary a3) conditional significant as with the polymers are higher, which are in the polymer dispersions of the state of the art present.

An other advantage the particular cross-linkingable N-methylol-haltigen compounds a2) is that that the crosslinking can become targeted effected by a thermal method step with the processing. Thus the polymer dispersion can be processed due to its low viscosity despite its high active substance content excellent, to z. B. lay on in thin layer on a surface. Only then the made thermal method step, z. B. Dry, if necessary bottom elevated temperature. The molecular weight increase by crosslinking of the polymerization film a conditional particularly good cohesion of the layer, taking place thereby. Therefore a main field of application of the erfindungsgeinässen polymer dispersions is the use as adhesive, z. B. as coating agents for wallpapers.

When diluting the aqueous polymer dispersion PD), PD min) as well as PD min min) the current viscosity rises to a very high maximum, whereby the system becomes clearer. Here the thickening action of the dispersed polymer A becomes) significant.

An other advantageous feature of the aqueous polymer dispersions according to invention is the high shearing and condition stability. Thus the high viscosity of an aqueous solution with 1% content at polymer remains A) even after longer agitation constant. This property is likewise favourable for the use as component of a wallpaper coating.

The absence of organic solvents an ensured safe handling (for example no flammability) and an ecological acceptable use of the polymer dispersions according to invention PD) PD min) and/or PD min min) as thickening agents, as flocculants for sewage sludge, as retention means for the papermaking and/or as soil improvement means. In isolated or water-poor form the Polymerisatgemisch according to invention can become as drainage means, for example within the hygiene range, used.

The subsequent examples are to describe the invention. The physical data became certain on the basis the subsequent norms:

- Dynamic viscosity η (mPa.s) according to DIN 53018/53019
 - Molecular weight M_w by gel permeation chromatography (see. z. B. H.F. Mark of et al., locomotive CIT., volume. 10, sides 1 to 19) with standard Poly-2-Trimethylammoniummethylacrylatchlorid.
 - Master mountain value STB II (s):
- Determination of the time course of kaolin sedimentation with Flockungsmittel haltigen solutions after the flocculation procedure.

Per liter tap water (20 DEG DH) 20 g kaolin suspended and bottom become agitations homogeneous held. Afterwards 250 becomes ml kaolin suspension in 250 ml-Messzylinder filled and bottom agitations an homogeneous held. To the dosage of 1 ml 0,1%iger of aqueous

solution of the polymer dispersion PD), PD min) and/or. PD min min) becomes the agitation interrupted.

Subsequent one becomes still 15 s agitated and from this the agitation stopped. Afterwards the time for the decrease of the sedimentation mirror becomes taken around 4 cms in the Messzylinder, which corresponds to the master mountain value STB II.

EXAMPLES

Example 1

Polymer dispersion with N-methylol-functional Methacrylatverbindung

350.0 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylatchlorid-Lösung, 3 g butyl acrylate, 0.48 g N-Methylolmethacrylamid and 279.5 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and. Subsequent ones become 0.04 g 2.2 min - Azobis [2 (2-imidazolin-2-yl) - propane) dihydrochlorid (AIP) dissolved in 0,36 g water added. After 1,5 hours bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant Polymerdisperion PD1) is characterized by the subsequent sizes. The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 70,300 \text{ mPa.s}$. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 1560 \text{ mPa.s}$. The flocculation value amounts to: STB II = 17.0 S.

Dry content: 35%.

The polymer dispersion is suitable good as flocculant, in addition, as adhesive with high adhesive force.

Example 2

Polymer dispersion with N-methylol-functional Methacrylverbindung, monomer addition in two stages

350.0 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylatchlorid-Lösung, 3 g butyl acrylate, 0.48 g N-Methylolmethacrylamid and 279.5 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and. Subsequent ones become 0.04 g 2.2 min - Azobis [2 (2-imidazoling-2-yl) - propane] dihydrochlorid (AIP) dissolved in 0,36 g water added. At the temperature temperature of the exothermic reaction (T_{max}) other 0.48 g become N-Methylolmethacrylamid added. After 1,5 hours bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 44,900$ mPa.s. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 1750$ mPa.s. The flocculation value amounts to: STB II = 14.4 S.

Dry content: 35%.

The polymer dispersion is suitable good as flocculant, in addition, as adhesive with high adhesive force.

Example 3

Polymer dispersion with N-methylol-functional Methacrylverbindung, addition in two stages

350.0 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylatchlorid-Lösung, 3 g butyl acrylate, 0.48 g N-Methylolmethacrylamid and 279.5 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and. Subsequent ones become 0.04 g 2.2 min - Azobis [2 (2-imidazolin-2-yl) - propane] dihydrochlorid (AIP) dissolved in 0,36 g water added. At the temperature temperature of the exothermic reaction (T_{max}) other 1.92 become g N-Methylolmethacrylamid added. After 1,5 hours agitation becomes the temperature on 65 degrees C elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 77,800$ mPa.s. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 2340$ mPa.s. The flocculation value amounts to: STB II = 12.6 S.

Dry content: 35%.

The polymer dispersion is suitable good as flocculant, in addition, as adhesive with high sticking detention.

Example 4

Polymer dispersion contained both a N-Methylolfunktionelle Methacrylsäureverbindung and monomer with two radical polymerizable, ethylenic unsaturated groups

350.0 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylatchlorid-Lösung, 3 g butyl acrylate, 0.48 g N-Methylolmethacrylamid, 20 ppm N, N min - Methylenbis (methacrylamid) and 279.5 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and. Subsequent ones become 0.04 g 2.2 min - Azobis [2 (2-imidazolin-2-yl) - propane] - dihydrochlorid (AIP) dissolved in 0,36 g water added. After 1,5 hours bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 554,000$ mPa.s. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 1670$ mPa.s. The flocculation value amounts to: STB II = 18.5 S.

Dry content: 35%.

The polymer dispersion is suitable good as flocculant.

Example 5

Polymer dispersion with N-methylol-functional Methacrylverbindung, weak controlled

350.0 g of a 40%igen Poly DADMAC solution, 194.0 g of a 50%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylatchlorid-Lösung, 3 g butyl acrylate, 4.32 g N-Methylolmethacrylamid, 0.1 g 2-Mercaptoethanol and 320.3 g waters become agitations on 55 degrees C heated, bottom in a Reaktionsgefäß with N₂ degassed and. 0.04 g 2.2 min - Azobis [2 (2-imidazolin-2-yl) - propane] - dihydrochlorid (AIP) dissolved in 0,36 g water added. After 1,5 hours bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer

dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 11,200$ mPa.s. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 126$ mPa.s. The flocculation value amounts to STB II = 33.0 S.

Dry content: 35%.

The polymerization is suitable particularly good as adhesive with high adhesive force.

Example 6

Polymer dispersion with N-methylol-functional Methacrylverbindung, strong controlled

350.0 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylchlorid-Lösung, 3 g butyl acrylate, 5.76 g N-Methylolmethacrylamid, 0.2 g 2-Mercaptoethanol and 269.3 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and.

Subsequent ones become 0.04 g 2.2 min - Azobis [2 (2-imidazolin-2-yl) - propane] - dihydrochlorid (AIP) dissolved in 0,36 g water added. After 1,5 h bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40% ig) diluted. The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 36,000$ mPa.s. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 11.2$ mPa.s. The flocculation value amounts to: STB II = 43.2 S.

Dry content: 35%.

The polymer dispersion is suitable particularly good as adhesive with high adhesive force.

Example 7

Polymer dispersion with N-methylol-functional acryl connection

350 g of a 40%igen Poly DADMAC solution, 242.5 g of a 40%igen acrylamide solution, 125.0 g of a 80%igen aqueous 2-Trimethylammoniummethacrylchlorid-Lösung, 3 g butyl acrylate, 0.1 g N-methylol acrylamide and 278.1 g waters become agitations on 55 degrees C heated, bottom in a reaction vessel with N₂ degassed and. Subsequent ones become 0.04 g 2.2 min -

Azobis [2 (2-imidazolin-2-yl) propane] dihydrochlorid (AIP) dissolved in 0,36 g water added. After 1,5 hours bottom agitations the temperature on 65 degrees C becomes elevated and other 0.2 g AIP dissolved in 1,8 g water added. The post-reaction lasts 1 hour with constant temperature from 65 degrees C. Subsequent one becomes the resultant polymer dispersion PD) with 200,0 g Poly DADMAC solution (40%ig) diluted.

The resultant polymer dispersion PD1) is characterized by the subsequent sizes: The dynamic viscosity of the aqueous polymer dispersion amounts to $\eta_1 = 53,100 \text{ mPa.s}$. The dynamic viscosity of a one percent aqueous solution of the high molecular polymer amounts to $\eta_2 = 1160 \text{ mPa.s}$. The flocculation value amounts to STB II = 13.5 S.

Dry content: 35%.

The polymer dispersion is suitable particularly good as adhesive with high adhesive force.



Claims of DE19532229

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1. Method to the production low-viscous, water-soluble polymer dispersions PD) on aqueous basis with high active substance concentrations, characterised in that the monomer components

a1) 50 to 99.99 Gew. - % at least water-soluble monomers,

a2) 0.01-5 Gew. - % of a cross-linkingable N-methylol-compound

a3) 0 to 1 Gew. - % at least crosslinking monomers with at least two ethylenic unsaturated, radical polymerizable groups, as well as if necessary

a4) 0 to 30.Gew. - % at least hydrophobic monomers and

a5) 0 to 20 Gew. - % at least amphiphilic monomers

▲ top. in aqueous solution in presence at least a polymere dispersing agent D) to a polymer A) polymerized become that itself the monomer components a1), a2), a3), a4) and a5) 100 Gew. - supplement % that the resultant polymer A) an average molecular weight Mw of at least 5×10^5 Dalton exhibits, as well as that the polymer A) with the dispersing agent D) incompatible is.

2. Method according to claim 1, characterised in that at least one water-soluble monomers the a1) an ionic group exhibits.

3.Verfahren in accordance with the claims 1 and 2, characterised in that the cross-linkingable N-methylol-compound a2) monomer with an ethylenic unsaturated radical polymerizable group, which possesses or several N-methylol-functional groups, is.

4. Method in accordance with the claims 1 and 2, characterised in that the cross-linkingable N-methylol-compound A) a not radical polymerizable compound is, which contains at least 2 condensationable N-methylol-groups.

5. Method according to claim 3, characterised in that a2) N-methylol (meth) acrylamide is.

6. Method in accordance with the claims 1 to 5, characterised in that crosslinking monomers the a3) selected is from the group that (meth) the acryl connections, that trichloroethylene (meth) acryl connections, that tetra (meth) acryl connections and/or (Meth) the allyl (meth) acryl connections.

7. Verfahren in accordance with the claims 1 to 6, characterised in that the monomer component a4) to 1 to 25 Gew. - is % at the formation of the polymer A) involved.

8. Method in accordance with the claims 1 to 7, characterised in that the monomer component a5) to 0.1 to 15 Gew % at the formation of the polymer involved is.

9. Method in accordance with the claims 1 to 8, characterised in that hydrophobic monomers the a4) a compound of the formula I is:

EMI40.1

how

G 1 for hydrogen or alkyl with 1 to 5 carbon atoms, as well as

R2 for alkyl, Cycloalkyl, aryl or Aralkyl with 1 to 20 carbon atoms or for

EMI40.2

with R3 for alkyl, Cycloalkyl, aryl or Aralkyl with 1 to 20 carbon atoms and Z for O, NH or NR3 to stand can.

10. method in accordance with the claims 1 to 9, characterised in that amphiphilic monomers the a5) a compound of the formulas II or IIa is

EMI41.1

how

A1 for O, NH, NR4 with R4 for alkyl with 1 to 4 carbon atoms,

R5 for hydrogen or methyl,

R6 for alkyls with 1 to 18 carbon atoms,

R7 and R8 independently for alkyl with 1 to 6 carbon atoms

R9 for alkyls with 1 to 6 carbon atoms,

R min 9 for alkyl with 1 to 18 carbon atoms,

R10 for alkyl, aryl or Aralkyl with 8 to 32 carbon atoms, as well as

X for halogen, OCN, SCN, SO4CH3 or acetate

to stand can.

11-method in accordance with the claims 1 to 9, characterised in that amphiphilic monomers the a5) a compound of the formulas III and IIIa is.

EMI41.2

EMI42.1

how

A2 for O, NH, NR13 with R13 for alkyl with 1 to 4 carbon atoms,

R11 for hydrogen or methyl

R12 for alkyl, aryl or Aralkyl with 8 to 32 carbon atoms,

R min 12 for hydrogen or alkyl with 1 to 4 carbon atoms,

R14 for alkyls with 1 to 18 carbon atoms

Y for alkyls with 2 to 6 carbon atoms as well as

n for a whole number between 1 and 50

to stand can.

12. Method in accordance with the claims 1 to 11, characterised in that the polymere dispersing agent D) a polyelectrolyte with an average molecular weight M_w of less than 5×10^5 Dalton is.

13. Verfahren in accordance with the claims 1 to 12, characterised in that to the production of the polymer A) in presence of the polymere dispersing agent D) in the first stage a second stage follows, in which the water-soluble polymer dispersion PD) other polymere dispersing agent D) in aqueous solution added becomes.

14. Method according to claim 13, characterised in that during the first stage of the production of the polymer A) in presence of the polymere dispersing agent D) and/or between the first and second stage method according to claim 13 the water content of the polymerization PD) reduced becomes.

15. Use by means of the methods in accordance with the claims of the 1 to 14 prepared polymer dispersions as flocculants for electric loaded suspended particles, as retention means for the papermaking, as thickening agents, as component of paper or wallpaper coatings, as drainage means and/or as soil improvement means.